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CONTAMINANT REMOVAL FROM PLATING BATHS BENCH-SCALE EVALUATION OF ELECTROLESS NICKEL BATH REJUVENATION VOLUME IV

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13. ABSTRACT (*Maximum 200 words*)

Electroless nickel (EN) plating is performed at all U.S. Air Force ALCs as part of depot level maintenance of aircraft parts. EN baths are frequently (once a month) dumped due to reaction byproduct (orthophosphite) build-up in the bath. Battelle was contracted by Armstrong Laboratory/Environics Directorate (AL/EQS) to identify, test, and implement a suitable technology to rejuvenate spent EN baths. After a technology review, three different technologies were considered for the rejuvenation of EN baths. After initial testing, one of them, the Stapleton Enfinity process, was selected for detailed bench scale testing. Plating tests with continuous bath rejuvenation were performed for 10 metal turnovers. Bath constituents were continuously monitored to determine the efficacy of orthophosphite (contaminant) removal from the bath. Plating quality, phosphorous content of deposit and deposit stress characteristics were analyzed and were found to meet the required specifications. Waste generated from the process (calcium orthophosphite filter cake) was collected and analyzed. The filter cake was successfully washed to reduce the nickel content to less than 5 ppm by TCLP. Alternate methods to monitor nickel content of the bath (in the presence of calcium) were developed. Plating rate, deposit characteristics, and waste generation were favorably compared to conventional EN processes. Based on results of these tests, it was recommend that a full-scale prototype unit of the Stapleton process with filter cake washing be designed, installed, and demonstrated at Tinker AFB OC-ALC.

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Electroless nickel plating, bath rejuvenation, orthophosphite removal from plating baths.

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PREFACE

This Final report represents the results of work done by Battelle in Columbus, Ohio, on Volume IV, "Economics of a New Electroless Nickel Plating Bath Rejuvenation Process" Contract No. F08635-90-C-0064, with the Environics Directorate, Armstrong Laboratory, Tyndall AFB, Florida.

This Final Report, covers February 1993 to June 1994. Our team's efforts were expanded by the conscientious involvement of others. We would like to express our appreciation to Glenn Graham, Tom Walker, Patti Shreve, Ernie Barlor, Danny Summrall, and Jerry Jones of the U.S. Air Force for lending their process experience and time. Their input was critical in identifying the true nature of the technical problems to overcome.

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EXECUTIVE SUMMARY

A. OBJECTIVE

This report provides the economic and feasibility analysis for the installation of Stapleton electroless nickel bath rejuvenation system at plating shops. The information in this report can be used by all USAF-Air Logistics Plating Shop personnel performing electroless nickel plating on aircraft parts.

B. BACKGROUND

Electroless Nickel (EN) plating is routinely performed at USAF-ALCs as a means of providing corrosion resistance to aircraft parts. EN plating is an autocatalytic chemical reaction with reaction byproducts accumulating in the bath. With usage, the byproducts accumulation slows the plating rate and renders the bath inoperable. Traditionally EN baths are dumped once a month, constituting a significant hazardous waste source from plating shops. Battelle tested and evaluated several bath rejuvenation (byproduct removal) technologies and selected Stapleton Enfinity process for prototype installation at Tinker AFB. The process selectively removes the bath reaction byproduct by selective precipitation using lime and eliminates the need for bath dumps. The only waste from this process is a nonhazardous calcium orthophosphite sludge.

C. SCOPE

This report briefly describes the bath rejuvenation technology and provides a complete economic analysis for the installation of a fullscale prototype at OC-ALC. The report discusses the installations costs, operating costs, waste generation and disposal costs and payback periods for the Stapleton EN bath rejuvenation system. Section I is an introduction to the conventional EN process and the problems associated with EN bath dumps. It also provides the approach to the economic analysis. Section II describes current EN operations at ALCs and the Stapleton bath rejuvenation technology. It compares the current operations with proposed technology and provides the material balances for current and proposed EN operations. Section III provides the economic analysis with details on capital and operating costs and the payback periods for the proposed Stapleton rejuvenation system. Section IV compares the waste generation from current and proposed EN plating operations. Conclusions and recommendations are given in Section V. Appendix A describes the cake-washing tests to remove trace nickel from the calcium orthophosphite filter cake.

D. CONCLUSIONS

Stapleton Enfinity EN bath rejuvenation system is an efficient way to remove EN bath contaminants and to eliminate bath dumps. Based on conservative assumptions, the installed cost of standard Stapleton system is projected to be $78,114 and the annual savings are projected to be $38,487 resulting in a payback period of 2 years. These results are based on a 300 gallons EN bath operating at a rate of 50 metal turnovers per year. Process variable sensitivity analysis indicates that the overall operating cost of electroless nickel plating is significantly affected by the chemical cost, plating rate (which affects the labor cost) and labor cost. In addition to the environmental benefits associated with the elimination of bath dumps and lower operating costs, the Stapleton process has the advantage of consistent plating quality due to unchanging bath composition. It is recommended that the Stapleton system be installed to replace the conventional EN process and, after gaining sufficient operating experience, the cake-washing system may be implemented, if needed.

TABLE OF CONTENTS

Section	Title	Page
I.	INTRODUCTION	1
	A. OBJECTIVE	1
	B. BACKGROUND	1
	C. APPROACH	2
II.	PROCESS DESCRIPTION	3
	A. ELECTROLESS NICKEL PLATING PROCESS	3
	B. CURRENT EN BATH OPERATION AT TINKER AFB	3
	C. STAPLETON ELECTROLESS NICKEL	6
	D. STAPLETON ELECTROLESS NICKEL WITH CAKE-WASHING	6
III.	ECONOMIC ANALYSIS	14
	A. EQUIPMENT, LABOR AND UTILITY COSTS	14
	B. CAPITAL COST	18
	C. OPERATING COSTS	22
	D. SENSITIVITY ANALYSIS AND PAY-BACK PERIOD	22
IV.	WASTE GENERATION AND DISPOSAL	29
V.	CONCLUSIONS AND RECOMMENDATIONS	31
Appendix		
A.	ELECTROLESS NICKEL FILTER CAKE-WASHING TESTS	33

LIST OF FIGURES

Figure	Title	Page
1.	Process Flowsheet for the Operation and Disposal of Existing Electroless Nickel Baths	4
2.	Process Flowsheet for the Standard Stapleton EN Process and Disposal	7
3.	Prototype of the Stapleton Electroless Nickel Bath Rejuvenation System	8
4.	Process Flowsheet for the Stapleton EN Process with Cake-Washing	10
5.	Process Flowsheet for the Stapleton EN Process with Acid Washing	12
6.	Net Annual Operating Costs for Conventional Electroless Nickel and Stapleton Electroless Nickel Processes	26
7.	Comparison of Wastes Generated from Conventional and Stapleton EN Processes	30

LIST OF TABLES

Table	Title	Page
1.	Material Balances for Conventional Electroless Nickel Process	5
2.	Material Balances for Standard Stapleton Electroless Nickel Process	9
3.	Material Balances for Stapleton Electroless Nickel Process Cake-Washing	11
4.	Material Balances for Stapleton Electroless Nickel Process with Acid Washing of Cake	13
5.	Electroless Nickel Economic Analysis - Process Assumptions	15
6.	Electroless Nickel Economic Analysis Model - Cost Assumptions	16
7.	Purchased Equipment Costs for Conventional Electroless Nickel	17
8.	Purchased Equipment Costs for Stapleton Electroless Nickel	17
9.	Purchased Equipment Costs for Stapleton Electroless Nickel with Cake-Washing	17
10.	Capital Cost Estimate for Conventional Electroless Nickel Process	19
11.	Capital Cost Estimate for Stapleton Electroless Nickel Process	20
12.	Capital Cost Estimate for Stapleton EN Process with Cake-Washing	21
13.	Net Annual Operating Cost for Conventional Electroless Nickel	23
14.	Net Annual Operating Cost for Stapleton Electroless Nickel	24
15.	Net Annual Operating Cost for Stapleton EN with Cake-Washing	25
16.	Process Variable Sensitivity Analysis and Payback Periods	28
A-1.	Nickel Content Data from Water Washing Tests	35
A-2.	TCLP Test Results for the Third Wash Cake	36
A-3.	Insoluble Nickel Content of Filter Cakes	37
A-4.	Estimate of Percent Pickup of Nickel in TCLP	38
A-5.	Simulation of Three-Stage Washing and Expected TCLP Results	41

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SECTION I
INTRODUCTION

The five Air Force Air Logistic Centers (AF-ALCs) carry out plating operations as part of their weapon systems overhaul and maintenance operations. During plating, a variety of contaminants, specially ionic species, accumulate in the plating baths and interfere with the plating process and degrade deposit characteristics. This leads to periodic dumping of the baths which constitute hazardous waste. To help alleviate this problem for the Air Force, the Environics Division of the Armstrong Laboratories contracted Battelle to carry out a research and development project, titled "Contaminant Removal from Plating Baths." The specific objective of this project was to develop separation technologies to remove contaminants from and thus rejuvenate two nickel plating baths: electroless nickel (EN) and nickel-strike (Ni-Strike).

The results from this project are reported in six volumes as follows:

Volume I.	Bench-Scale Evaluation of Electroless Nickel Bath Rejuvenation
Volume II.	Bench-Scale Evaluation of Nickel-Strike Bath Rejuvenation
Volume III.	Economics of a New Nickel-Strike Bath Rejuvenation Process
Volume IV.	Economics of a New Electroless Nickel Bath Rejuvenation Process
Volume V.	Electroless Nickel Bath Rejuvenation Prototype Demonstration
Volume VI.	Nickel-Strike Bath Rejuvenation Pilot Plant Demonstration.

This volume (No. IV) covers the economic analysis evaluation of electroless nickel bath rejuvenation processes.

A. OBJECTIVE

The objective of the work reported in this volume was to perform an economic analysis for the implementation of the selected electroless nickel bath rejuvenation system.

B. BACKGROUND

This report provides the economic and feasibility analysis for electroless nickel bath rejuvenation system. Electroless nickel (EN) plating is performed at Tinker AFB and the baths are dumped approximately once a month. EN bath wastes are difficult to treat and constitute significant source of hazardous waste from plating shops. As part of the Environics contract, Battelle undertook to test and evaluate methods to rejuvenate electroless nickel baths and eliminate the need for bath

dumps and their subsequent disposal. Battelle tested and evaluated various technologies and selected Stapleton Technologies system for EN bath rejuvenation. The tests and evaluation are summarized in an earlier report, "Bench-Scale Evaluation of a Process for Rejuvenation of Electroless Nickel Baths" (Volume I).* Subsequent to the testing and process selection, economic and feasibility analysis was performed on the selected technology and this report provides the details of the economic analysis and feasibility analysis.

The overall goal of project is to eliminate hazardous waste generation from the selected plating processes. Since the filter cake generated from EN bath rejuvenation has trace amounts of nickel, washing tests were conducted (after the process tests and evaluation) on bench scale and full-scale systems. These tests are described in detail in the appendix and test results are summarized in this report.

C. APPROACH

The economic analysis compares the existing process at Tinker AFB with the envisioned process that incorporates the EN bath rejuvenation. The approach followed for the comparison of these processes consisted of the following steps.

(1) Development of process flowsheets and material balances

(2) Specification of process equipment including materials of construction and sizes.

(3) Determination of purchased equipment costs and estimation of direct costs including installation

(4) Estimation total fixed capital investment including engineering and supervision and working capital

(5) Estimation of annual operating costs based on chemical supplies, labor, utilities, waste disposal and depreciation.

Based on the above approach, a mathematical cost model was developed for each process configuration. Comparison of annualized operating costs from the model show the economic viability of the proposed EN rejuvenation processes.

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* See Volume I of report on this project to Environics Directorate, August 12, 1993.

SECTION II

PROCESS DESCRIPTION

A. ELECTROLESS NICKEL PLATING PROCESS

EN plating is an autocatalytic reaction between nickel ions and hypophosphite ions in an aqueous solution. The desired product is the nickel-phosphorous deposit on the parts and the byproduct is the orthophosphite ion which accumulates in the bath. In traditional EN baths, consumed nickel and hypophosphite ions are replenished as aqueous solutions of nickel sulfate and sodium hypophosphite. EN bath life is measured in metal turnovers. One metal turnover (MTO) is when nickel metal equivalent to all the nickel originally in the bath has been plated on the parts. In EN plating, due to orthophosphite byproduct accumulation, the plating rate decreases and plating characteristics change and eventually the baths are dumped. EN bath dumps are difficult to waste treat because of the presence of many complexing agents in solution.

B. CURRENT EN BATH OPERATION AT TINKER AFB

At present, EN baths are dumped after 4 MTOs at Tinker AFB. After 4 MTOs of plating, the plating deposit changes from being compressively stressed to being tensile stressed, an unacceptable feature for aircraft parts at Tinker AFB. When an EN plating bath is discarded, it is pretreated with sodium hydroxide (NaOH) to bring the pH up to 10. At that pH, all the nickel in solution precipitates as nickel hydroxide which is filtered using a filter press and further dewatered in an oven. The nickel hydroxide sludge is then disposed of (offsite) as hazardous sludge at a cost of $4.25/kg ($1.94/lb). The filtrate containing all the complexing agents, accumulated orthophosphite and sulfate (total of 150,000 ppm) is slowly bled into the on-site industrial waste water treatment (IWTP) facility. This orthophosphite is eventually precipitated and itself disposed as a hazardous sludge at a cost of $4.25/kg.

Tinker AFB operates two 150-gallon EN plating baths. Process flowsheet for one 300-gal EN bath operation and disposal as presently practiced ("Conventional Electroless Nickel") is given in Figure 1. Material Balances for the process at an operating rate of 50 MTOs/year are given in Table 1. Stream numbers identified in Figure 1 correspond to the stream numbers in Table 1.

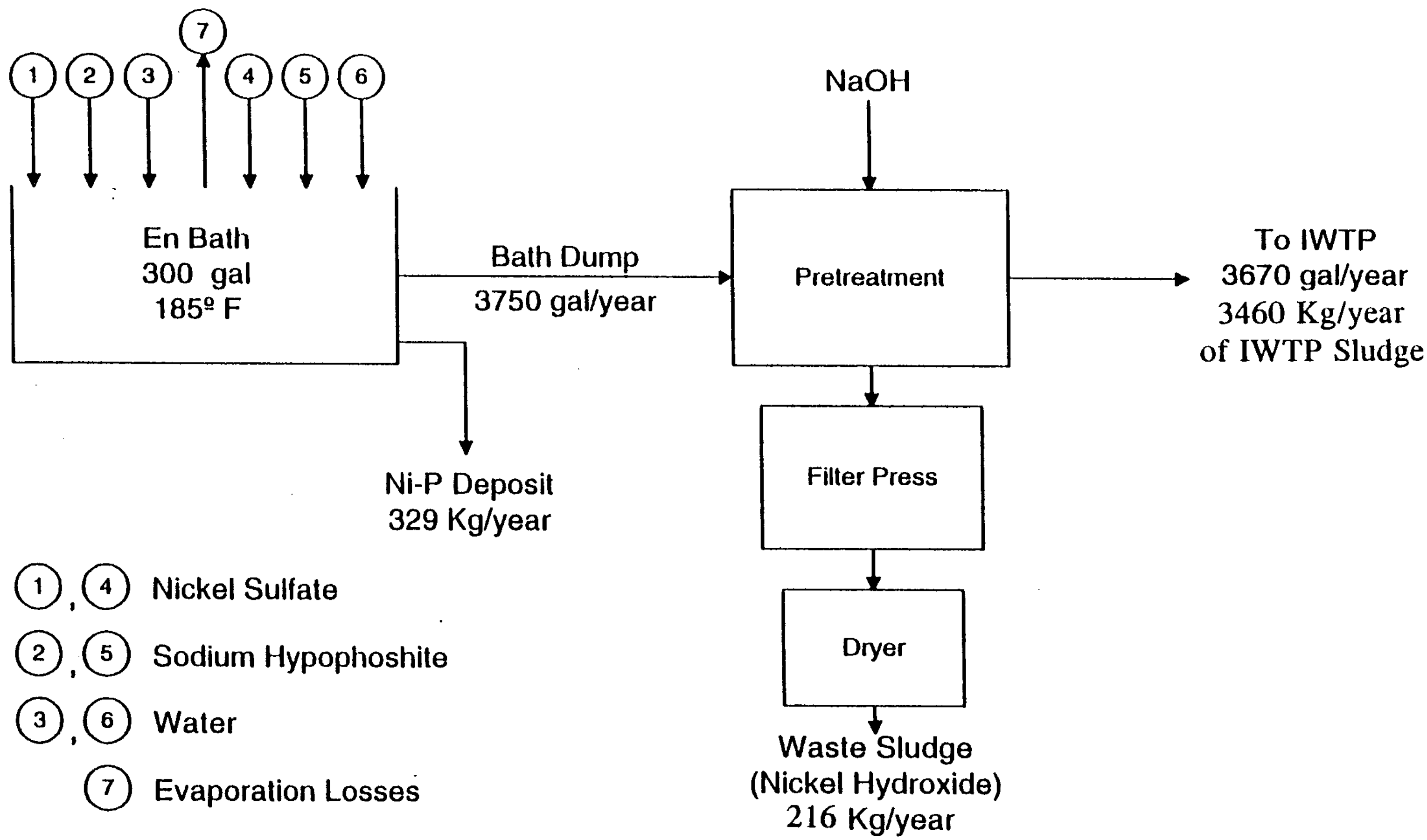

Figure 1. Process Flowsheet for the Operation and Disposal of Existing Electroless Nickel Baths.

TABLE 1. MATERIAL BALANCES FOR CONVENTIONAL ELECTROLESS NICKEL PROCESS

Stream No.	Stream Description	Quantity				Frequency		Yearly Quantity		Purchase Cost			Disposal Cost,		
		gal/MTO	gal/dump	kg/MTO	kg/dump	dumps/yr	MTO/yr	gal	kg	$/gal	$/kg	$/yr	$/kg	$/gal	$/yr
1	NiSO4 (300-A)		18			12.5		225		16.28		3,663			
2	NaH2PO2.H2O (300-B)		54			12.5		675		18.62		12,569			
3	Water		228			12.5		2,850		1.6E-03		4			
4	NiSO4 (300-A)	18					50	900		16.28		14,652			
5	NaH2PO2.H2O (300-D)	36					50	1,800		18.35		33,030			
6	Replenish Water	258					50	12,900		1.6E-03		20			
7	Water Evap. Loss	312					50	15,600		NA		NA			
8	Bath Dump		300			12.5		3,750		NA		NA			
9	Plated Nickel			6.59			50		329.3	NA		NA			
10	NaOH				54.35	12.5			679.4		2.88	1,957			
11	Sludge				294.13	12.5			3676.6			NA	4.25		15,626
12	Waste Water		245.6			12.5		3,070				NA		0.013	40
	.														
											SUM	$65,895		SUM	$15,666

NA: NOT APPLICABLE

C. STAPLETON ELECTROLESS NICKEL

The selected technology for rejuvenating electroless nickel baths is supplied by Stapleton Technologies, Long Beach, California. The Stapleton process has modified chemistry. Consumed nickel and hypophosphite are replenished as an aqueous solution of nickel hypophosphite and the only accumulating byproduct is the orthophosphite anion. The orthophosphite is removed by treating a slipstream from the bath with calcium hydroxide to precipitate calcium orthophosphite which is filtered out. The process flowsheet for the Stapleton electroless nickel is shown in Figure 2. A photograph of the Stapleton EN full-scale prototype unit is shown in Figure 3. Material balance of all streams are given in Table 2 for an annual operating rate of 43.61 MTOs/year of Stapleton EN bath (equivalent to 50 MTOs/year of conventional EN). Stapleton EN bath has a higher concentration of nickel in the bath and, consequently, requires less MTOs of plating than a conventional EN bath for an equivalent amount of plating deposit.

D. STAPLETON ELECTROLESS NICKEL WITH CAKE-WASHING

Stapleton EN process produces calcium orthophosphite filter cake, which has some amount of nickel in it due to the presence of residual plating liquid in the cake. Although nickel is not now regulated metal, it is anticipated that the EPA will regulate nickel in the future. Then the presence of trace amounts of nickel will render the filter cake a hazardous material. As per information from Tinker AFB's EM office, the expected limit on nickel is 5 ppm as determined by the Toxicity Characteristic Leaching Procedure (TCLP). In order to generate only nonhazardous waste from the Stapleton EN process, cake-washing was performed to reduce the nickel content of the cake below the (anticipated) 5 ppm TCLP limit. Bench-scale batch-washing tests were conducted in the laboratory to test the washing concept and full-scale bath-washing tests were conducted by Battelle at Stapleton Technologies in Long Beach, California. These tests are described in detail in Appendix A, "Electroless Nickel Filter Cake-Washing Tests." Based on the results of the batch-washing tests, a three-stage countercurrent water washing system was designed to reduce the nickel content of the cake with the wash water being returned to the bath. The flowsheet for the cake-washing is given in Figure 4 and the material balances are given in Table 3.

Alternatively, the three-stage water wash can be replaced with one-stage acid wash using sulfuric acid. The acid wash converts calcium orthophosphite to calcium sulfate and generate wastewater containing phosphorous acid which is sent to the IWTP. The acid-washing system removes practically all the nickel in the cake as soluble nickel sulfate. The flowsheet for the acid-washing system is given in Figure 5 and the corresponding material balances are given in Table 4.

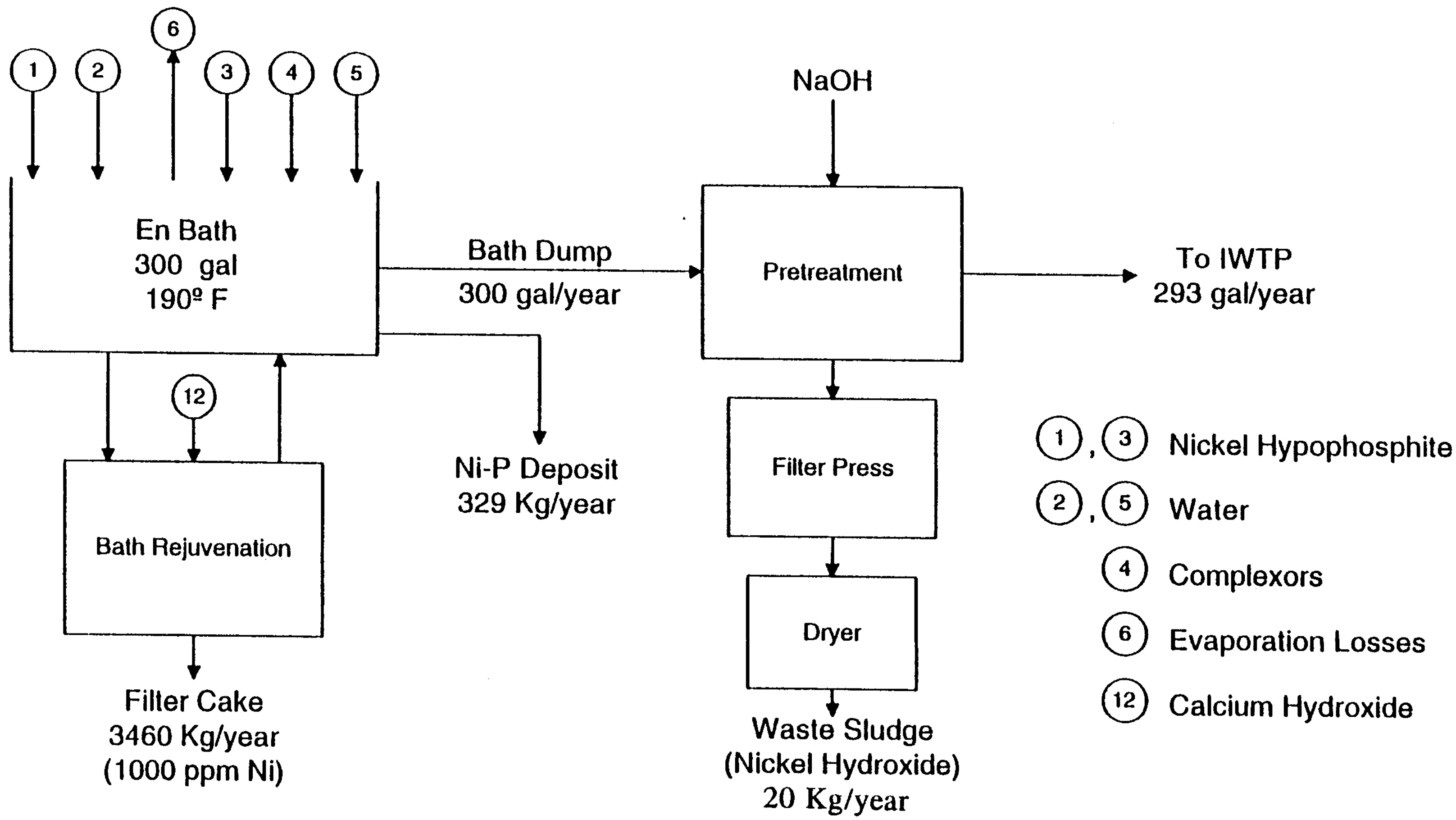

Figure 2. Process Flowsheet for the Standard Stapleton EN Process and Disposal.

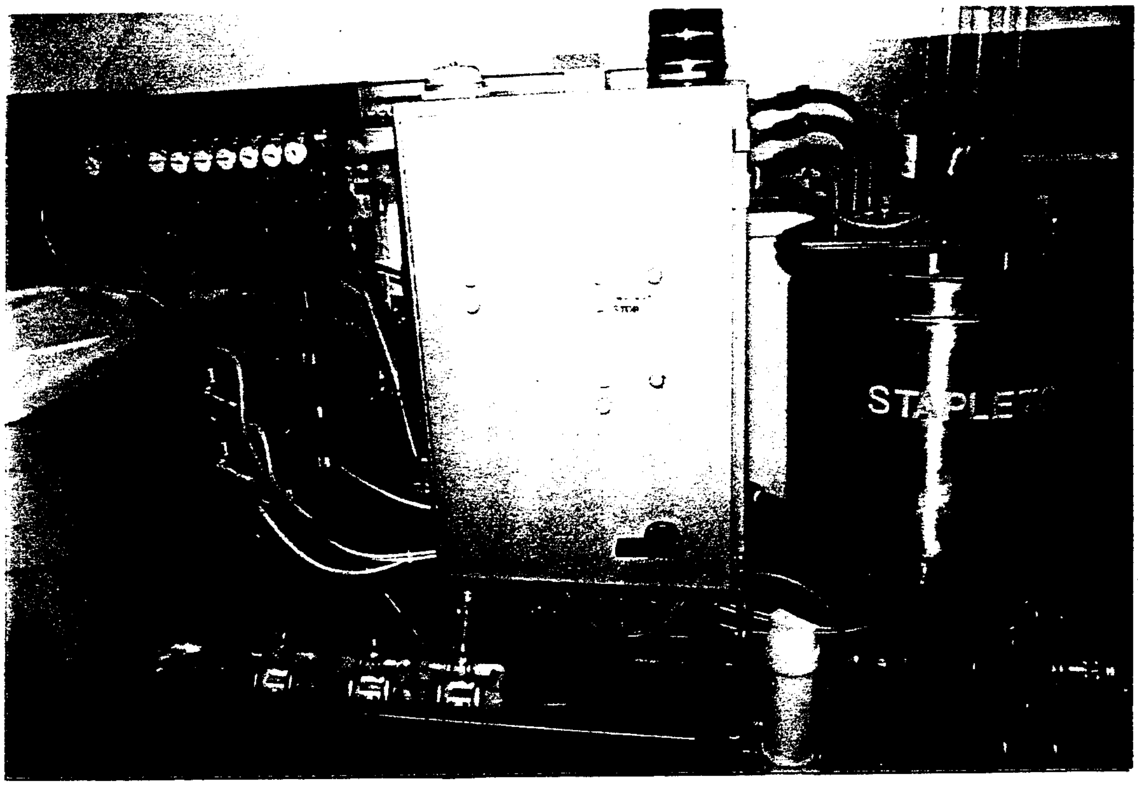

Figure 3. Prototype of the Stapleton Electroless Nickel Bath Rejuvenation System.

TABLE 2. MATERIAL BALANCES FOR STANDARD STAPLETON ELECTROLESS NICKEL PROCESS

Stream No.	Stream Description	Quantity				Frequency		Yearly Quantity		Purchase Cost			Disposal Cost,		
		gal/MTO	gal/dump	kg/MTO	kg/dump	dumps/yr	MTO/yr	gal	kg	$/gal	$/kg	$/yr	$/kg	$/gal	$/yr
1	NiHypophosphite (HXIA)		150			1		150.0		10.12		1,518			
2	Process Water		150			1		150.0		0.00155		0			
3	NiHypophosphite (HXIR)	76.6					43.6	3340.5		17.16		57,322			
4	NiHypophosphite (HXIC)	3					43.6	130.8		15.75		2,061			
5	Process Water	226.8					43.6	9890.5		0.00155		15			
6	Water Loss	294					43.6	12800.0		NA		NA			
7	Plated Nickel			7.55			43.6		329.3	NA		NA			
8	Bath Dump		300			1		300.0		NA		NA			
9	NaOH				55.66	1			55.7		2.88	160			
10	Sludge				19.85	1			19.9			NA	4.25		84
11	Waste Water		292.7			1		292.7				NA		0.013	4
12	Ca(OH)2			29.4			43.6		1282.1		1.76	2,257			
13A	Filter Cake (dry solids)			47.6			43.6		2075.8				4.25		8,822
13B	Filter Cake (water)	6.8					43.6	296.5					4.25		4,778
13C	Filter Cake (HXIR)	1.6					43.6	69.8					4.25		1,124
											SUM	$63,333		SUM	$14,813

NA: NOT APPLICABLE

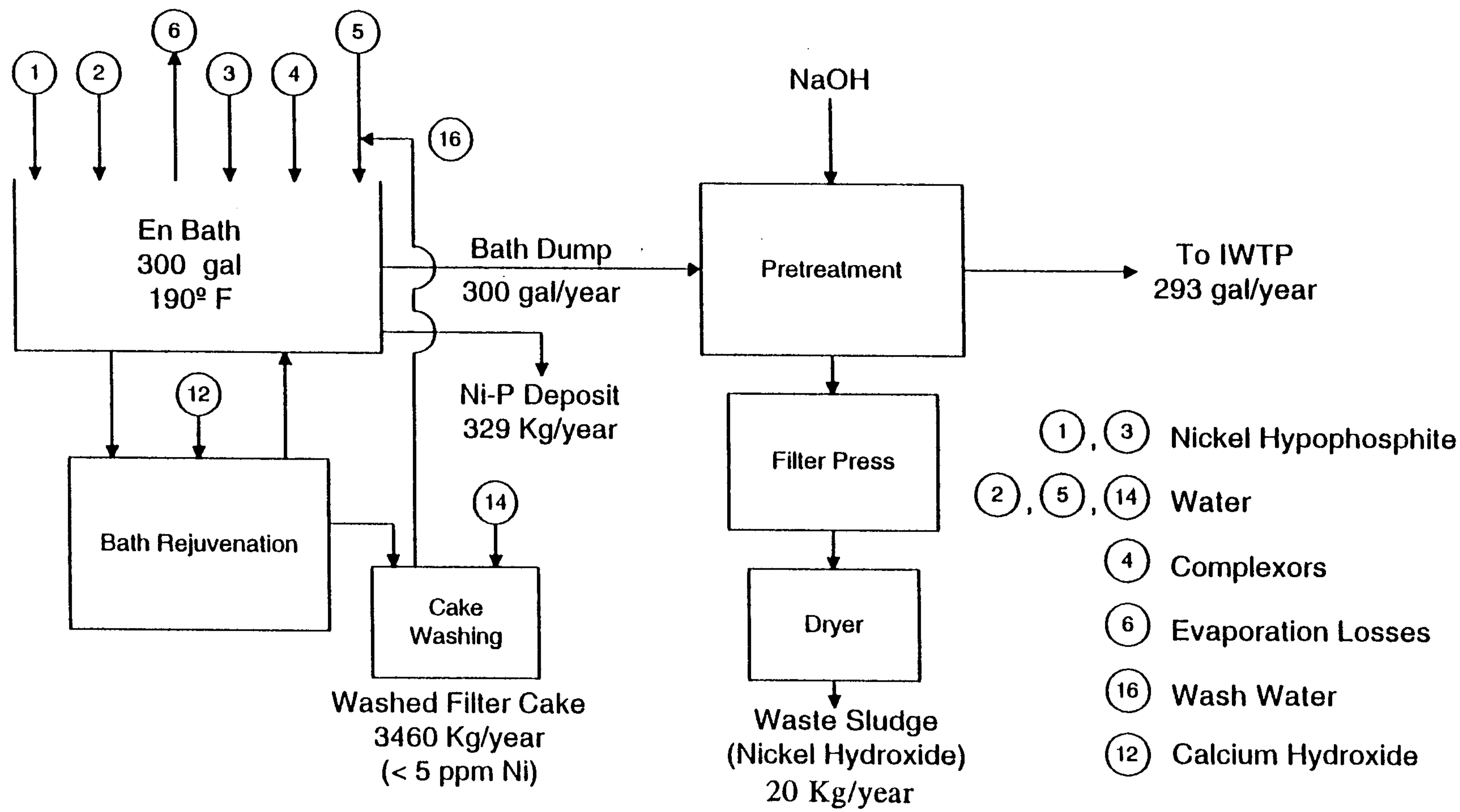

Figure 4. Process Flowsheet for the Stapleton EN Process with Cake Washing

TABLE 3. MATERIAL BALANCES FOR STAPLETON ELECTROLESS NICKEL PROCESS CAKE WASHING

Stream No.	Stream Description	Quantity gal/MTO	gal/dump	kg/MTO	kg/dump	Frequency dumps/yr	MTO/yr	Yearly Quantity gal	kg	Purchase Cost $/gal	$/kg	$/yr	Disposal Cost, $/kg	$/gal	$/yr
1	NiHypophosphite (HXIA)		150			1		150.0		10.12		1,518			
2	Process Water		150			1		150.0		0.00155		0			
3	NiHypophosphite (HXIR)	75					43.6	3270.7		17.16		56,125			
4	NiHypophosphite (HXIC)	3					43.6	130.8		15.75		2,061			
5	Process Water	165.4					43.6	7212.9		0.00155		11			
6	Water Loss	294					43.6	12800.0		NA		NA			
7	Plated Nickel			7.55			43.6		329.3	NA		NA			
8	Bath Dump		300			1		300.0		NA		NA			
9	NaOH				55.66	1			55.7		2.88	160			
10	Sludge				19.85	1			19.9			NA	4.25		84
11	Waste Water		292.7			1		292.7				NA		0.013	4
12	Ca(OH)2			29.4			43.6		1282.1		1.76	2,257			
13	Filter Cake			79.35			43.6		3460.4			NA			
14	Wash Water	63					43.6	2747.37		0.00155		4			
15A	Filter Cake (dry solids)			47.6			43.6		2075.8			NA	1.92		3,986
15B	Filter Cake (liquid)	8.4					43.6	366.3				NA	1.92		2,667
16	Filtrate/Wash Water	63					43.6	2747.4				NA			
											SUM	$62,136		SUM	$6,740

NA: NOT APPLICABLE

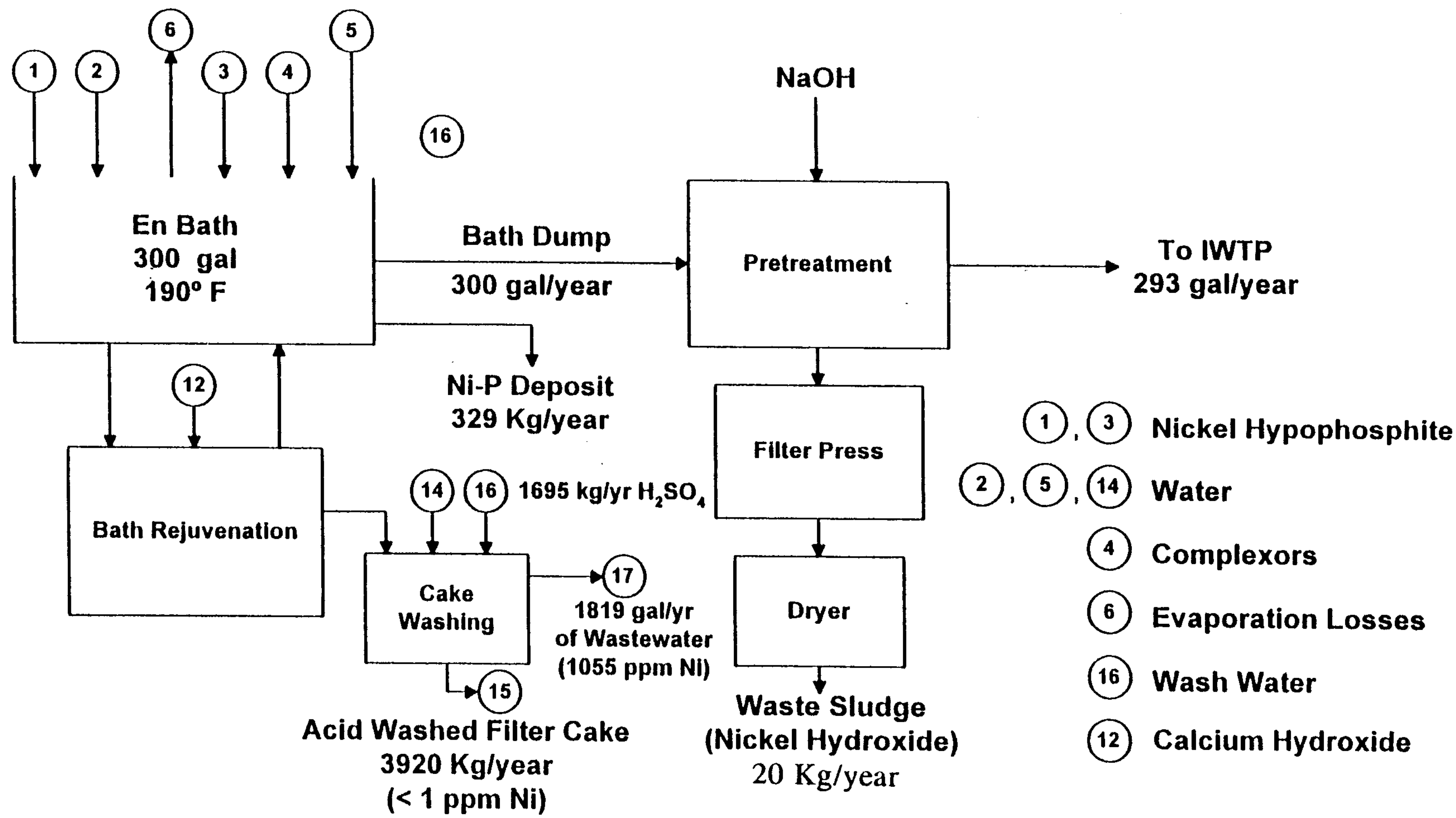

Figure 5. Process Flowsheet for the Stapleton EN Process with Acid Washing

TABLE 4. MATERIAL BALANCES FOR STAPLETON ELECTROLESS NICKEL PROCESS WITH ACID WASHING OF CAKE

Stream	Stream	Quantity				Frequency		Yearly Quantity		Purchase Cost			Disposal Cost,		
No.	Description	gal/MTO	gal/dump	kg/MTO	kg/dump	dumps/yr	MTO/yr	gal	kg	$/gal	$/kg	$/yr	$/kg	$/gal	$/yr
1	NiHypophosphite (HXIA)		150			1		150.0		10.12		1,518			
2	Process Water		150			1		150.0		0.00155		0			
3	NiHypophosphite (HXIR)	75					43.6	3270.7		17.16		56,125			
4	NiHypophosphite (HXIC)	3					43.6	130.8		15.75		2,061			
5	Process Water	165.4					43.6	7212.9		0.00155		11			
6	Water Loss	294					43.6	12800.0		NA		NA			
7	Plated Nickel			7.55			43.6		329.3	NA		NA			
8	Bath Dump		300			1		300.0		NA		NA			
9	NaOH				55.66	1			55.7		2.88	160			
10	Sludge				19.85	1			19.9			NA	4.25		84
11	Waste Water		292.7			1		292.7				NA		0.013	4
12	Ca(OH)2			29.4			43.6		1282.1		1.76	2,257			
13	Filter Cake			79.35			43.6		3460.4			NA			
14	Wash Water	63					43.6	2747.4		0.00155		4			
15A	Filter Cake (dry solids)			53.96			43.6		2353.1			NA	1.92		4,518
15B	Filter Cake (liquid)	9.5					43.6	414.3				NA	1.92		3,016
16	H2SO4			38.87			43.6		1695.1		0.08	136			
17	Filtrate/Wash Water	63					43.6	2747.4				NA			
											SUM	$62,271		SUM	$7,622

NA: NOT APPLICABLE

SECTION III
ECONOMIC ANALYSIS

An economic analysis was carried out to compare the operating and capital costs of the current EN process at Tinker AFB with three variations of the Stapleton EN process; (a) Standard Stapleton EN process (b) Stapleton EN process with water washing of the filter cake, and (c) Stapleton EN process with acid washing of the filter cake. A cost model was used to perform the economic analysis. Several process assumptions were made in the cost model and these are listed in Table 5. The process assumptions such as the bath size, MTOs/year and MTOs/bath dump correspond to the existing values at Tinker AFB (300-gallon bath, 50 MTOs/year and bath dump rate of every 4 MTOs respectively). Although the bath never needs to be dumped in the Stapleton process, a conservative assumption of dumping the bath once a year was assumed for economic analysis. Several cost assumptions were made in the cost model as shown in Table 6. These include chemical reagent costs, waste disposal cost and utilities cost. The assumed costs correspond to the present costs for these items. The other major cost constituent is the labor cost which is taken as $15/hour.

A. EQUIPMENT, LABOR AND UTILITY COSTS

Tables 7, 8, and 9 represent the purchased equipment cost, labor cost and energy cost for conventional EN, Stapleton EN and Stapleton EN with cake-washing, respectively. Water washing or acid washing of the cake required the same equipment and labor as given in Table 9. Although no equipment is purchased for conventional EN, for comparison purposes, costs have been assigned to the existing equipment at Tinker AFB. Stapleton process has one major equipment cost; i.e., the rejuvenation system. Stapleton EN with cake-washing has additional auxiliary equipment cost for the countercurrent washing such as holding tanks and an additional filter. Energy usage expressed as hp-hours per year are comparable for all processes at around 80,000 hp hours/year. Labor costs are substantially higher for conventional EN. This is a direct consequence of higher plating rate for the Stapleton process chemistry whose plating rate of 350 microinches/hr is 50 percent higher than the rate for conventional EN processes (230 microinches/hr). The plating rate values for the Stapleton process are a conservative estimate from the bench-scale tests and the plating rate values for the convention EN process are the average plating rate at Tinker AFB during 4 MTOs of plating (performed between 1/30/95 and 3/6/95).

TABLE 5. ELECTOLESS NICKEL ECONOMIC ANALYSIS - PROCESS ASSUMPTIONS

No.	Assumptions	
1	Batch size, gal	300
	Batch area, ft2	13
2	Operating factor, wk/yr	50
3	Conventional EN metal turnover, MTO/wk	1
4	Conventional EN MTO/yr	50
5	Conventional EN Ni conc, g/l	5.80
	Nickel plated, g/yr	329,295
6	Stapleton conc., g/l	6.65
	Stapleton MTO/yr	43.61
7	Bath load, ft2/gal	0.1
8	Conventional EN plating rate, micro-in/hr	230
9	Nominal Ni content in deposit	90.00%
10	Ni-P density, g/ml	8
	Conventional system required hr of operation, hr/	2,809
11	Operating hr contingency	8%
	Conventional EN operating hr/yr	3,000
12	Stapleton plating rate, micro-in/hr	350
	Stapleton operating hr/yr	1,971
13	Conventional EN, MTO per batch	4
	Conventional EN bath dumps/yr	12.5
14	Stapleton bath dumps/yr	1
15	Conventional EN water loss rate, gal/ft2/hr	0.4
16	Stapleton water loss rate, gal/ft2/hr	0.5
17	Conventional EN water loss, gal/yr	15600
	Conventional EN water loss, gal/MTO	312
18	Stapleton water loss, gal/yr	12800
	Stapleton water loss, gal/MTO	294
19	Wash water requirements, kg/kg wet cake	3
20	Stapleton filter cake % solids	60%
21	Stapleton washed filter cake % solids	60%

TABLE 6. ELECTROLESS NICKEL ECONOMIC ANALYSIS MODEL - COST ASSUMPTIONS

No.	COST ASSUMPTIONS		
22	NiSO4 (300-A)	$16.28	per gal
23	NaH2PO2.H2O (300-B)	$18.62	per gal
24	NaH2PO2.H2O (300-D)	$18.35	per gal
25	NaOH	$4.20	per kg
26	PROCESS WATER	$1.55	per 1000 gal
27	SLUDGE, HAZ. DISPOSAL	$4.25	per kg
28	SLUDGE, NON HAZ DISPOSAL	$1.92	per kg
29	WASTE WATER DISPOSAL	$13.00	per 1000 gal
30	ELECTRIC POWER	$0.06	per kWhr
31	LABOR	$15.00	per man-hr
32	Nickel Hypophosphite (HXIA) INITIAL	$10.12	per gal
33	Nickel Hypophosphite (HXIR) REPLENISH	$17.16	per gal
34	Nickel Hypophosphite (HXIC) CONTROL	$15.75	per gal
35	Ca(OH)2	$1.76	per kg
36	H2SO4	$0.08	per kg

TABLE 7. PURCHASED EQUIPMENT COSTS FOR CONVENTIONAL ELECTROLESS NICKEL

SYMBOL	NAME	CAPACITY PER UNIT	NO. OF UNITS	HP PER UNIT	ENERGY CONSUMPTION: UNIT OPERATING FACTOR (HP/OPER HR)	(HR/DUMP)	MAT'L OF CONST.	TEMP., F	RESIDENCE TIME OR TIME FOR TANK DUMP/FILL	TIME UNITS	PURCHSED EQUIPMENT 2ND QTR 94 COST, $	NET HP-HR PER YR	WORKER HR PER OPER. HR	WORKER HR PER TANK DUMP	MAN-HR/YR
B-1	EN BATH WITH AGITATOR AND FILTER	300 gal	1	25	1		SS	182		HR	$6,000	70,223	1.33		4,000
PT-1	PRE-TREATMENT TANK WITH AGITATOR	200 gal	1	1		2	SS	182	6	HR	$1,500	25		6	75
FP-1	FILTER PRESS	4 gpm	1	5		2	SS	150	2	HR	$3,000	25		2	25
										SUM	$10,500	70273			4,100

TABLE 8. PURCHASED EQUIPMENT COSTS FOR STAPLETON ELECTROLESS NICKEL

SYMBOL	NAME	CAPACITY PER UNIT	NO. OF UNITS	HP PER UNIT	ENERGY CONSUMPTION: UNIT OPERATING FACTOR (HP/OPER HR)	(HR/DUMP)	MAT'L OF CONST.	TEMP., F	RESIDENCE TIME OR TIME FOR TANK DUMP/FILL	TIME UNITS	PURCHSED EQUIPMENT 2ND QTR 94 COST, $	NET HP-HR PER YR	WORKER HR PER OPER. HR	WORKER HR PER TANK DUMP	MAN-HR/YR
B-1	EN BATH WITH AGITATOR AND FILTER	300 gal	1	25	1		SS	190		HR	$6,000	49,286	1.33		2,629
PT-1	PRE-TREATMENT TANK WITH AGITATOR	200 gal	1	1		2	SS	190	6	HR	$1,500	2		6	6
FP-1	FILTER PRESS	4 gpm	1	5		2	SS	150	2	HR	$3,000	10		2	2
S-1	STAPLETON SYSTEM	300 gal	1	10	0.5		SS/PLASTIC	120		HR	39,200	9,857	0		0
										SUM	$49,700	59,155			2,637

TABLE 9. PURCHASED EQUIPMENT COSTS FOR STAPLETON ELECTROLESS NICKEL WITH CAKE WASHING

SYMBOL	NAME	CAPACITY PER UNIT	NO. OF UNITS	HP PER UNIT	ENERGY CONSUMPTION: UNIT OPERATING FACTOR (HP/OPER HR)	(HR/DUMP)	MAT'L OF CONST.	TEMP., F	RESIDENCE TIME OR TIME FOR TANK DUMP/FILL	TIME UNITS	PURCHASED EQUIPMENT 2ND QTR 94 COST, $	NET HP-HR PER YR	WORKER HR PER OPER. HR	WORKER HR PER TANK DUMP	MAN-HR/YR
B-1	EN BATH WITH AGITATOR AND FILTER	300 gal	1	25	1		SS	190			6,000	49,286	1.33		2,629
PT-1	PRE-TREATMENT TANK WITH AGITATOR	200 gal	1	1		2	SS	190	6	HR	1,500	2		6	6
FP-1	FILTER PRESS	4 gpm	1	5		2	SS	150	2	HR	3,000	10		2	2
S-1	STAPLETON SYSTEM	300 gal	1	10	0.5		SS/PLASTIC	120			39,200	9,857	0		0
F-1	WASH FILTER	4 gpm	1	9	1		SS/PLASTIC	100			8,000	17,743	0.25		0
HT-1,3	HOLDING TANKS	50 gal	3	0	0		PLASTIC	100			990	0			493
MT-1	MIX TANK	50 gal	1	0.2	0.1		SS	100			1,000	39			0
P-1,3	TRANSFER PUMPS	2 gpm	3	1	0.1		SS	100			1,050	197			0
C-1	CONTROLLER		1	0	0			70			500	0			0
V-1,5	CONTROL VALVES		5	0	0		PLASTIC	70			500	0			0
															0
										SUM	$61,740	77,134			3,129

B. CAPITAL COST

Total capital costs for conventional EN, Stapleton EN and Stapleton EN with cake-washing are given in Tables 10, 11 and 12, respectively. As mentioned earlier, Stapleton EN with acid washing of cake needs the same equipment as the water washing process. Hence, the capital costs requirements are the same as the Stapleton EN process with water washing of cake (Table 12). Total capital cost includes direct plant costs (purchased equipment, installation, piping and instrumentation), indirect costs such as engineering and supervision as well as working capital and start-up costs. Equipment for conventional EN consists of individual units (plating tank, pretreatment tank, filter press) that need to be installed and connected. The direct and indirect costs were computed as a function of purchased equipment cost as is done for chemical plant capital cost estimates*. The Stapleton EN and Stapleton EN with cake-washing are purchased as assembled, prepackaged skid-mounted units (Figure 3). For these, the installation, piping, electrical and instrumentation costs are small and the actual or anticipated costs were used in the capital cost estimates. For all three processes, working capital is taken as 3 months of chemical inventory. The start-up capital cost for conventional EN is taken as zero since it is an existing process. The start-up capital costs for the Stapleton processes were taken as one month of operating labor and overhead in order to account for the process familiarization by the operators. The total capital costs for all EN processes (plating 329 kg of nickel-phosphorous deposit a year) are summarized below.

	Conventional EN (current)	**Stapleton EN**	**Stapleton EN with Cake-Washing**
Fixed Capital Investment	$30,550	$78,114	$96,801
(Dollars/kg Ni Plated)	($93)	($237)	($294)
Total Capital Investment	$46,528	$100,279	$119,876
(Dollars/kg Ni Plated)	($141)	($305)	($364)

* Peter and Timmerhaus, Plant Design and Economics for Chemical Engineers, Fourth Edition, 1991.

TABLE 10. CAPITAL COST ESTIMATE FOR CONVENTIONAL ELECTROLESS NICKEL PROCESS

COST ITEM	COST, $	BASIS
PURCHASED EQUIPMENT	10,500	100.00% OF PE COST
P.E. INSTALLATION	4,095	39.00% OF PE COST
INSTRUMENTATION AND CONTROL	1,365	13.00% OF PE COST
PIPING	2,625	25.00% OF PE COST
ELECTRICAL	1,050	10.00% OF PE COST
BUILDING	0	0.00% OF PE COST
YARD IMPROVEMENTS	0	0.00% OF PE COST
SERVICE FACILITIES	0	0.00% OF PE COST
LAND	0	0.00% OF PE COST
STORAGE	0	
TOTAL DIRECT PLANT COST	$19,635	187.00% OF PE COST
ENGINEERING AND SUPERVISION	3,360	32.00% OF PE COST
CONSTRUCTION EXPENSE	3,570	34.00% OF PE COST
TOTAL DIRECT & INDIRECT COSTS	$26,565	253.00% OF PE COST
CONTRACTORS FEES	1,328	5.00% OF DIRECT & INDIRECT COSTS
CONTINGENCY	2,657	10.00% OF DIRECT & INDIRECT COSTS
FIXED CAPITAL INVESTMENT	$30,550	
$/kg Ni PLATED	$93	
WORKING CAPITAL	15,978	3 MONTHS STOCK OF CHEMICALS
STARTUP COST	0	NO START-UP COST FOR EXISTING PROCE
TOTAL CAPITAL INVESTMENT	$46,528	
$/kg Ni PLATED	$141	

3,000 OPERATING hr/yr

TABLE 11. CAPITAL COST ESTIMATE FOR STAPLETON ELECTROLESS NICKEL PROCESS

COST ITEM	COST, $	BASIS
PURCHASED EQUIPMENT	49,700	100.00% OF PE COST
P.E. INSTALLATION	4,800	120 hrs OF CONTRACT LABOR, $40/hr
INSTRUMENTATION AND CONTROL	1,000	PURCAHSE COST OF COLORIMETER
PIPING	2,485	5.00% OF PE COST
ELECTRICAL	2,485	5.00% OF PE COST
BUILDING	0	0.00% OF PE COST
YARD IMPROVEMENTS	0	0.00% OF PE COST
SERVICE FACILITIES	0	0.00% OF PE COST
LAND	0	0.00% OF PE COST
STORAGE	0	
TOTAL DIRECT PLANT COST	$60,470	122.00% OF PE COST
ENGINEERING AND SUPERVISION	4,970	10.00% OF PE COST
CONSTRUCTION EXPENSE	2,485	5.00% OF PE COST
TOTAL DIRECT & INDIRECT COSTS	$67,925	137.00% OF PE COST
CONTRACTORS FEES	0	0.00% OF DIRECT & INDIRECT COSTS
CONTINGENCY	10,189	15.00% OF DIRECT & INDIRECT COSTS
FIXED CAPITAL INVESTMENT	$78,114	
$/kg Ni PLATED	$237	
WORKING CAPITAL	15,789	3 MONTHS STOCK OF CHEMICALS
STARTUP COST	6,377	1 MONTH OF LABOR AND OVERHEAD
TOTAL CAPITAL INVESTMENT	$100,279	
$/kg Ni PLATED	$305	

1,971 OPERATING hr/yr

TABLE 12. CAPITAL COST ESTIMATE FOR STAPLETON EN PROCESS WITH CAKE WASHING

COST ITEM	COST, $	BASIS
PURCHASED EQUIPMENT	61,740	100.00% OF PE COST
P.E. INSTALLATION	6,000	150 hrs OF CONTRACT LABOR, $40/hr
INSTRUMENTATION AND CONTROL	1,000	PURCHASE COST OF COLORIMETER
PIPING	3,087	5.00% OF PE COST
ELECTRICAL	3,087	5.00% OF PE COST
BUILDING	0	0.00% OF PE COST
YARD IMPROVEMENTS	0	0.00% OF PE COST
SERVICE FACILITIES	0	0.00% OF PE COST
LAND	0	0.00% OF PE COST
STORAGE	0	
TOTAL DIRECT PLANT COST	$74,914	121.00% OF PE COST
ENGINEERING AND SUPERVISION	6,174	10.00% OF PE COST
CONSTRUCTION EXPENSE	3,087	5.00% OF PE COST
TOTAL DIRECT & INDIRECT COSTS	$84,175	136.00% OF PE COST
CONTRACTORS FEES	0	0.00% OF DIRECT & INDIRECT COSTS
CONTINGENCY	12,626	15.00% OF DIRECT & INDIRECT COSTS
FIXED CAPITAL INVESTMENT	$96,801	
$/kg Ni PLATED	$294	
WORKING CAPITAL	15,490	3 MONTHS STOCK OF CHEMICALS
STARTUP COST	7,585	1 MONTH OF LABOR AND OVERHEAD
TOTAL CAPITAL INVESTMENT	$119,876	
$/kg Ni PLATED	$364	

1,971 OPERATING hr/yr

C. OPERATING COSTS

Annual operating costs for plating 329.3 kg of nickel phosphorous deposit are given for conventional EN process, Stapleton EN process and Stapleton EN process with cake-washing are given in Tables 13, 14, and 15, respectively. Operating costs include:

- Raw materials (chemicals)
- Labor (operating, maintenance and supervision)
- Plant overhead
- Depreciation
- Waste disposal
- Utilities (water and electricity).

The operating costs were estimated on the basis of a 300-gallon EN bath operating at 50 MTOs/year (43.6 MTOs/year for Stapleton EN) and producing a Ni-P deposit of 329.3 kg/year. The operating cost per kilogram of Ni-P deposit for the three processes are \$635, \$518, and \$546 respectively with both versions of Stapleton EN process having lower costs than conventional EN.

The total operating costs and the distribution of various cost items for all the three processes are compared in Figure 6. In all cases, labor and plant overhead and raw material cost account for a major portion (80 to 90 percent) of the total operating cost. Because of the faster plating rate of the Stapleton EN process (50 percent faster than conventional EN), significant savings are achieved in labor and overhead costs relative to conventional EN. As mentioned earlier, the plating rate value of 350 microinches/hr for the Stapleton process is based on the bench-scale tests for 10 MTOs. The conventional EN plating rate value is from the actual average plating rate at Tinker AFB during 4 MTOs of plating (starting with a fresh bath on 1/30/95 and continuing onto the eventual dumping of the bath on 3/6/95). The raw material costs are roughly equal for all three processes. It should be noted that waste disposal costs are a small portion of the overall operating costs and do not play a significant role in economic analysis. This is partly due to the high chemical and labor costs of EN plating and also due to lack of significant difference in the disposal of hazardous waste and nonhazardous waste. This waste generation and disposal costs are discussed further in the Waste Generation section, below.

D. SENSITIVITY ANALYSIS AND PAY-BACK PERIOD

Standard Stapleton EN process with bath rejuvenation requires a fixed capital investment of \$78,114. The operating cost difference between conventional and Stapleton EN processes is \$117/kg

TABLE 13. NET ANNUAL OPERATING COST FOR CONVENTIONAL ELECTROLESS NICKEL

COST ITEM	COST, $	BASIS	% OF OPER.COST
RAW MATERIALS			
NiSO4 (300-A)	18,315	$16.28 per gal	8.76%
NaH2PO2.H2O (300-B)	12,569	$18.62 per gal	6.01%
NaH2PO2.H2O (300-D)	33,030	$18.35 per gal	15.79%
LABOR			
OPERATING	61,500	$15.00 per hr	29.41%
MAINTENANCE	916	3.00% OF FCI	0.44%
SUPERVISION	9,225	15.00% OF OPERATING LABOR	4.41%
PLANT OVERHEAD	42,985	60.00% OF OPER AND MAINT.	20.55%
TANK-DUMP SLUDGE DISPOSAL	15,626	$4.25 per kg	7.47%
WASTE WATER TREATMENT	40	$13.00 per 1000 gal	0.02%
SLUDGE TREATMENT (NaOH)	1,957	$2.88 per kg	0.94%
OPERATING SUPPLIES	3,075	5.00% OF OPERATING LABOR	1.47%
MAINTENANCE SUPPLIES	1,222	4.00% OF FCI	0.58%
LABORATORY CHARGES	3,075	5.00% OF OPERATING LABOR	1.47%
UTILITIES			
ELECTRICITY	3,144	$0.06 per kWhr	1.50%
PROCESS WATER	4	$1.55 per 1000 gal	0.00%
DEPRECIATION	2,444	8.00% OF FCI	1.17%
TOTAL ANNUAL COSTS	$209,127		100.00%
$/kg, Ni PLATED	$635		

TABLE 14. NET ANNUAL OPERATING COST FOR STAPLETON ELECTROLESS NICKEL

COST ITEM	COST, $	BASIS	% OF OPER COST
RAW MATERIALS			
Nickel Hypophosphite (HXIA) INITIAL	1,518	$10.12 per gal	0.89%
Nickel Hypophosphite (HXIR) REPLENISH	57,322	$17.16 per gal	33.59%
Nickel Hypophosphite (HXIC) CONTROL	2,061	$15.75 per gal	1.21%
Ca(OH)2	2,257	$1.76 per kg	1.32%
LABOR			
OPERATING	39,549	$15.00 per hr	23.18%
MAINTENANCE	2,343	3.00% OF FCI	1.37%
SUPERVISION	5,932	15.00% OF OPERATING LABOR	3.48%
PLANT OVERHEAD	28,695	60.00% OF OPER AND MAINT.	16.82%
TANK-DUMP SLUDGE DISPOSAL	84	$4.25 per kg	0.05%
STAPLETON SLUDGE DISPOSAL	14,725	$4.25 per kg	8.63%
WASTE WATER TREATMENT	4	$13.00 per 1000 gal	0.00%
SLUDGE TREATMENT (NaOH)	160	$2.88 per kg	0.09%
OPERATING SUPPLIES	1,977	5.00% OF OPERATING LABOR	1.16%
MAINTENANCE SUPPLIES	3,125	4.00% OF FCI	1.83%
LABORATORY CHARGES	1,977	5.00% OF OPERATING LABOR	1.16%
UTILITIES			
ELECTRICITY	2,647	$0.06 per kWhr	1.55%
PROCESS WATER	15	$1.55 per 1000 gal	0.01%
DEPRECIATION	6,249	8.00% OF FCI	3.66%
TOTAL ANNUAL OPERATING COSTS	170,640		100.00%
$/kg, Ni PLATED	518		

TABLE 15. NET ANNUAL OPERATING COST FOR STAPLETON EN WITH CAKE WASHING

COST ITEM	COST, $	BASIS	% OF OPER COST
RAW MATERIALS			
NiHypophosphite (HXIA)	1,518	$10.12 per gal	0.84%
NiHypophosphite (HXIR)	56,125	$17.16 per gal	31.24%
NiHypophosphite (HXIC)	2,061	$15.75 per gal	1.15%
Ca(OH)2	2,257	$1.76 per kg	1.26%
LABOR			
OPERATING	46,941	$15.00 per hr	26.13%
MAINTENANCE	2,904	3.00% OF FCI	1.62%
SUPERVISION	7,041	15.00% OF OPERATING LABOR	3.92%
PLANT OVERHEAD	34,132	60.00% OF OPER AND MAINT.	19.00%
TANK-DUMP SLUDGE DISPOSAL	84	$4.25 per kg	0.05%
STAPLETON SLUDGE DISPOSAL	6,652	$1.92 per kg	3.70%
WASTE WATER TREATMENT	4	$13.00 per 1000 gal	0.00%
SLUDGE TREATMENT (NaOH)	160	$2.88 per kg	0.09%
OPERATING SUPPLIES	2,347	5.00% OF OPERATING LABOR	1.31%
MAINTENANCE SUPPLIES	3,872	4.00% OF FCI	2.16%
LABORATORY CHARGES	2,347	5.00% OF OPERATING LABOR	1.31%
UTILITIES			
ELECTRICITY	3,451	$0.06 per kWhr	1.92%
PROCESS WATER	15	$1.55 per 1000 gal	0.01%
DEPRECIATION	7,744	8.00% OF FCI	4.31%
TOTAL ANNUAL OPERATING COSTS	179,656		100.00%
$/kg, Ni PLATED	546		

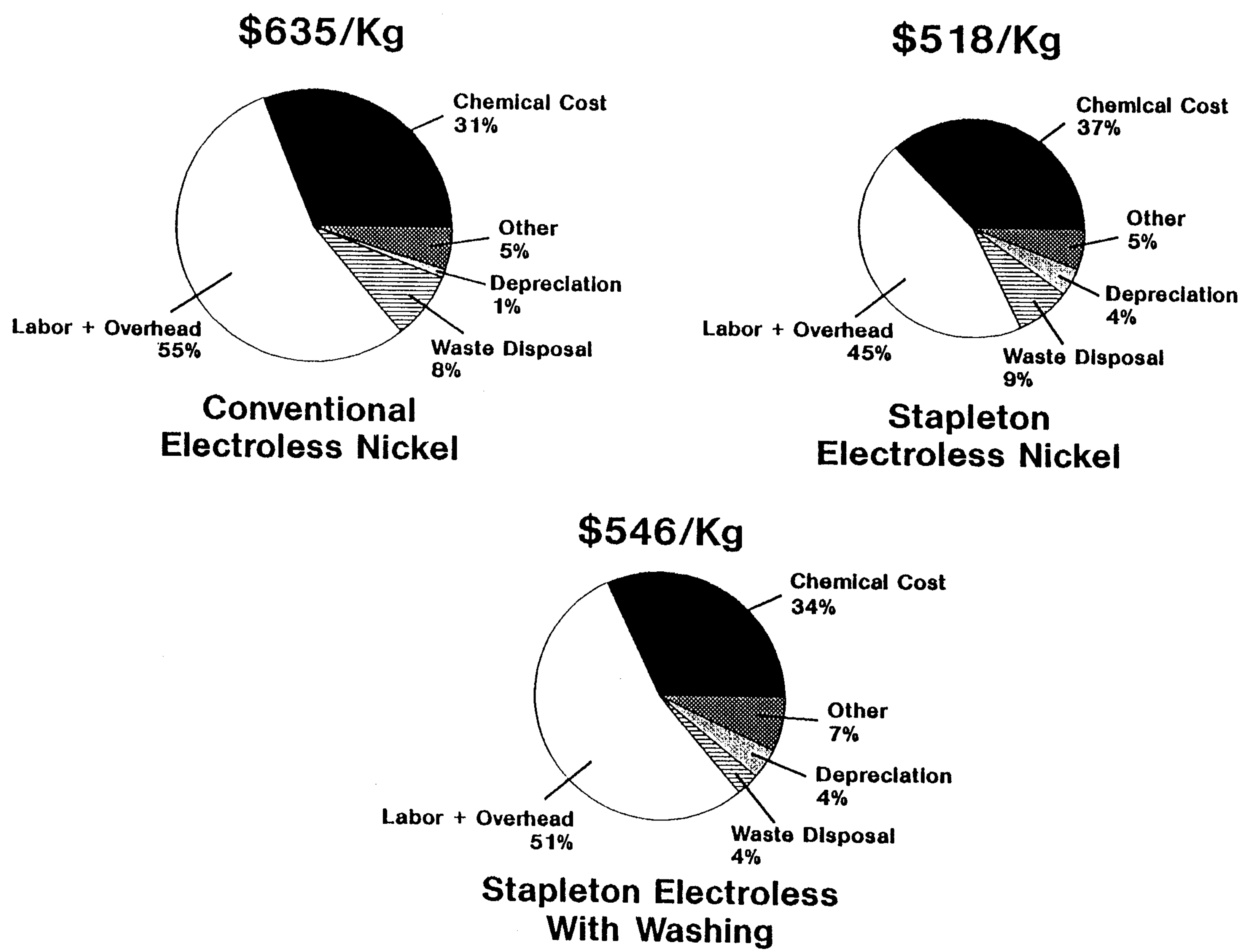

Figure 6. Net Annual Operating Costs for Conventional Electroless Nickel and Stapleton Electroless Nickel Processes

of Ni-P deposit (\$635 - \$518). The annual savings in operating cost would be \$38,487. The savings correspond to a pay-back period of 2 years. In addition, Stapleton processes have the added benefit of consistent plating quality because of uniform bath composition over a prolonged period (more than a year). The decision to deploy that process should also consider the added environmental benefit of almost complete elimination of hazardous waste from EN processes, as well as the benefit of avoiding risks and costs associated with managing the waste disposal.

In arriving at these annual savings and payback period, it was assumed that when a conventional EN bath is dumped and waste treated, the orthophosphite containing waste water is sent to the on-site IWTP. At the IWTP, it is assumed that the orthophosphite ion will precipitate as sludge and eventually disposed as hazardous IWTP (F006) sludge. The reason for this assumption is based on the solubility of orthophosphite ion in the presence of Ca^{++} ion in solution. At a pH of 8 (IWTP conditions), the solubility of orthophosphite is estimated to be 1 ppm in the presence of 1000 ppm of Ca^{++}, and 10 ppm in the presence of 100 ppm of Ca^{++} ion. Since lime addition is practiced at the IWTP, it is reasonable to expect the presence of Ca^{++} ion and hence the precipitation of phosphite ion as sludge. It is estimated that this contributes 276.8 kg/bath dump of sludge (60 percent solids).

Although the economic analysis was performed for the Stapleton system with cake-washing (see Tables 4, 9, 12 and 15), later meetings with OC-ALC plating shop and process engineering personnel indicated that they would not initially implement a washing system. Their approach was to implement to the standard Stapleton rejuvenation system, gain experience, analyze the filter cake and then consider the washing system. Hence, the payback period and sensitivity analysis (given below) compares only the Stapleton system (without cake-washing) with a conventional EN process.

The operating costs of EN plating using either the conventional process or the Stapleton process are sensitive to:

- plating rate which effects the labor cost
- labor cost
- waste disposal costs
- chemical costs.

The base values (for these variables) used in the economic analysis are given in Tables 5 and 6. The variation in operating costs and payback periods due to changes in these base values has been estimated and summarized in Table 16.

TABLE 16. PROCESS VARIABLE SENSITIVITY ANALYSIS AND PAYBACK PERIODS

Process Variable	Variable Value	Annual Operating Cost, $/kg N		Payback Period
		Conventional EN Process	Stapleton EN Process	
Single Sided Plating Rate for Conventional EN	0.23 mils/hr (Base)	635	518	2yrs.
	0.20 mils/hr	695	518	1yr 4mos.
	0.25 mils/hr	611	518	2yrs 8mo
Labor Cost	$15/man-hr (Base)	635	518	2yrs.
	$20/man-hr	756	596	1yr 6mos.
	$25/man-hr	877	674	1yr 2mos.
Stapleton Chemical Cost	80% of Base Cost	635	481	1yr 7mos.
	120% of Base Cost	635	555	3yrs.
Hazardous Waste Disposal Cost	$4.25/kg (base)	635	518	2yrs.
	$2.12/kg	611	495	2yrs.
	$1.06/kg	599	484	2yrs.

SECTION IV

WASTE GENERATION AND DISPOSAL

Conventional EN baths are dumped after four MTOs. They are then neutralized with alkali which generates nickel hydroxide sludge and wastewater containing dissolved orthophosphite, hypophosphite, sulfate and sodium. The sludge is disposed as hazardous waste and the wastewater containing dissolved orthophosphite is discharged to the IWTP. Stapleton processes generate calcium orthophosphite filter cake. Theoretically, the Stapleton bath need never be dumped. However, to be conservative, we estimated one bath dump per year. This bath dump produces small amounts of wastewater and sludge. The calcium orthophosphite filter cake can be rendered nonhazardous by washing and removing the trace amount of nickel in the cake. Figure 7 compares the annual waste generated from conventional EN, Stapleton EN and Stapleton EN with cake-washing.

As shown in Figure 7, all EN processes produce waste. However, they are different in content. Stapleton process generates filter cake with trace amounts of nickel. Conventional EN produces nickel hydroxide sludge and wastewater containing orthophosphite. The wastewater from conventional EN (3670 gallons/year) contains 150,000 ppm of dissolved orthophosphite, sulfate and hypophosphite. Since lime addition is practiced in the IWTP, the orthophosphites in the wastewater precipitate and generate sludge equivalent to the Stapleton EN process and an additional cost is incurred in disposing the sludge from IWTP as hazardous waste.

One of the goals of this project is to reduce the nickel discharge to the environment from electroless nickel plating. In conventional EN plating, when a bath is dumped after 4 MTOs, nickel is discharged as hazardous sludge (nickel hydroxide). This nickel discharge amounts to 25 percent of the nickel added to the bath. In the Stapleton process, the bath is not dumped and the only nickel discharge to the environment is in the trace amounts of nickel in the filter cake. Based on bench-scale tests and prior full scale tests, nickel discharge in a standard Stapleton process is 1.5 percent of the nickel feed resulting in a nickel utilization of 98.5 percent versus 75 percent for a conventional bath. Nickel is one of the seventeen compounds in the EPA's "33/50" program whose discharge has to be reduced by 33 percent by 1992 and 50 percent by 1995 based on 1991 discharge levels. The Stapleton process effectively reduces the nickel discharge from En operations by 95 percent simply by eliminating the periodic bath dumps.

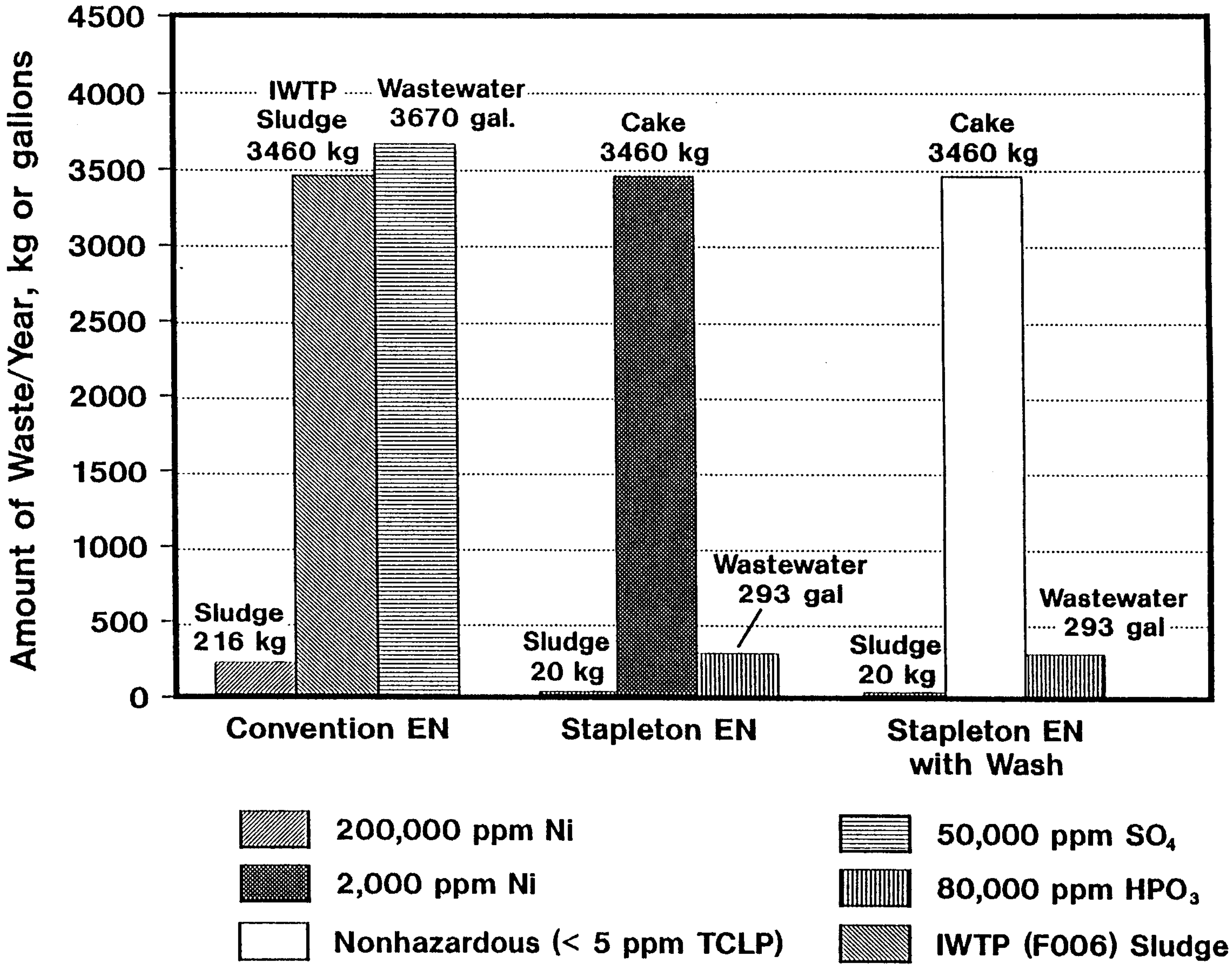

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Figure 7. Comparison of Wastes Generated from Conventional and Stapleton EN Processes.

SECTION V

CONCLUSIONS AND RECOMMENDATIONS

Economic analysis of Stapleton EN process is discussed in this report. Material balances, capital cost estimates, and operating cost estimates were obtained for the Stapleton EN process with bath rejuvenation and compared to the conventional EN process with bath disposal. The analyses show:

1. Approximately $78,000 of fixed capital investment is needed to implement the Stapleton EN process and $96,800 of fixed capital investment is needed for Stapleton EN process with cake-washing.
2. The annual operating cost of plating 329 kg of Ni-P deposit per year in a 300 gallons bath is $209,000, $170,000 and $179,000 for conventional EN, Stapleton EN and Stapleton EN with cake-washing respectively.
3. Pay-back period of 2 years is estimated to recoup the fixed capital investment for the Stapleton EN Process with bath rejuvenation.
4. Process variable sensitivity analysis shows that the overall cost of electroless nickel plating is significantly affected by chemical costs, plating rate (which affects labor costs) and labor cost.

Based on our analysis, we recommend that Standard Stapleton Process be installed to replace conventional EN process. After gaining sufficient operating experience, the cake-washing system may be implemented.

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APPENDIX A

Electroless Nickel Filter Cake-Washing Tests

Appendix A

ELECTROLESS NICKEL FILTER CAKE-WASHING TESTS

Stapleton Enfinity process rejuvenates the electroless nickel bath by removing the bath's primary contaminant, orthophosphite, as calcium orthophosphite. The calcium orthophosphite is removed as a filter cake and it constitutes the only waste generated by the process. Although most of the filtrate (containing nickel and hypophosphite values) is returned to the bath, some filtrate adheres to the calcium orthophosphite crystals, resulting in a nickel contaminated cake. Although nickel is not a regulated metal for waste disposal at present, it is expected that EPA will regulate nickel in the near future. It is expected that the future limits on nickel content in industrial sludges will be 5 ppm as determined by Toxic Characteristic Leaching Procedure (TCLP).

One of the primary goals of the project on electroless nickel bath rejuvenation is reduction in the generation of hazardous waste. Hence, tests were carried out by washing the cake in multiple (three) stages with water to reduce its nickel content. The cake was filtered after every wash and reslurried with fresh water. The cake from the final (third) wash was tested for its nickel content using TCLP. The filter cake-washing tests, analysis and results are described below.

A. FILTER CAKE-WASHING TESTS

A total of eight washing tests were conducted to determine the efficacy of washing to remove nickel from the filter cake. Of these three were conducted at Battelle on the filter cake obtained from the benchscale electroless nickel (EN) bath rejuvenation tests performed at Battelle. The other five tests were conducted by Battelle on the filter cake from bath rejuvenation tests on the fullscale prototype unit (PU2) performed at Stapleton Technologies, Long Beach, California. Of these five, the first three consisted of only three-stage water washers and the last two were two-stage water washes followed by an acid wash. In each case, the filter cake is first weighed and its moisture content is determined. Then it is slurried with water so that the water to wet cake mass ratio is approximately 3 to 1. The slurry is filtered again and the process is repeated twice more. For the two acid wash tests, during the reslurrying of the third wash, concentrated sulfuric acid was added until the pH became 1.5. After every filtration, the cake and filtrate samples are taken and the moisture content of the cake, as well as the nickel content of the cake and filtrate, is determined by

inductively coupled argon plasma technique (ICAP). Finally, the cake from the 3rd wash is sent to TCLP analysis. The solids percent of the cake and nickel content of the cake and filtrate values from the six water wash tests are given in Table A-1. The TCLP test results for the third wash cake for all the eight tests are given in Table A-2.

TABLE A-1. NICKEL CONTENT DATA FROM WATER WASHING TESTS.

Test No.	Sample I.D.	Cake Solids (percent)	Nickel in Wet Cake (ppm)	Nickel in Filtrate (ppm)	Comment
1	B1W0 B1W1 B1W2 B1W3	64.6 35.4 27.9 35.2	2466 800 300 300	 868 194 103	TCLP = 6.32
2	B2W0 B2W1 B2W2 B2W3	58.0 42.8 38.8 43.0	1890 746 326 229	 839 192 135	TCLP = 6.66
3	B3W0 B3W1 B3W2 B3W3	71.1 42.0 33.3 43.9	4270 770 328 214	 1040 274 81	3rd wash with 1% EDTA solution TCLP = 4.19
4	BST1W0 BST1W1 BST1W2 BST2W3	59.1 60.2 58.5 57.6	1897 1296 1236 1134	 218 80 11	TCLP = 9.56
5	BST2W0 BST2W1 BST2W2 BST2W3	62.8 52.3 54.2 62.7	2516 1773 1581 1494	 279 94 23	0th cake rinsed with 0.5% ammonia solution TCLP = 17.1
6	BST3W0 BST3W1 BST3W2 BST3W3	62.4 55.0 49.9 52.5	2308 1300 1157 1044	 252 58 29	1st wash contained 150 ml of glacial acetic acid. TCLP = 13.2

* The last digit on the sample indicates the wash number. Zeroth wash is the filter cake from the initial calcium orthophosphite filtration.

TABLE A-2. TCLP TEST RESULTS FOR THE THIRD WASH CAKE.

Test No.	Sample I.D.	TCLP Value (ppm nickel)	Comment
1	B1W3	6.32	
2	B2W3	6.66	
3	B3W3	4.19	3rd wash is with 1% EDTA solution
4	BST1W3	9.56	
5	BST2W3	17.1	
6	BST3W3	13.2	
7	BST4AW3	0.87	3rd wash is with acid, pH=1.5
8	BST5AW3	1.38	3rd wash is with acid, pH=1.5

As shown in Table A-2, the acid washing of filter cake clearly removes most of the nickel from the cake. Then the cake can be disposed of as nonhazardous material because the TCLP nickel content is below the 5 ppm limit. However, the cake-washing with only water is on the borderline of the 5 ppm limit, with the TCLP test results varying from 4.19 ppm to as high as 17.1 ppm. Plain water washing is preferable because the wash liquid can be recycled to the EN bath. In order to assess the reasons for failing the TCLP limit of 5 ppm, the data from the water washing tests were analyzed further. The data analysis is described below.

B. DATA ANALYSIS OF WATER WASHING TESTS

Acid washing of cake reacts with the nickel in the cake to form soluble nickel sulfate which is then removed in the filtrate. On the other hand, water washing can only remove soluble nickel in the cake. Nickel (Ni) in solid form as nickel metal or a nickel compound such as a hydroxide are not affected by the water washing. Based on the data of nickel content of the filtrate and the wet cake and the cake's moisture content, the amount of insoluble nickel in the wet cakes can be calculated as:

Insoluble Ni in cake = (Total Ni in cake) - (cake moisture percent x Ni in filtrate)

The only assumption made in the above equation is that the liquid fraction of the cake has the same nickel content as the filtrate. Table A-3 shows the insoluble nickel content of the filter cakes for the six water washing tests. The insoluble nickel content in the cake on a dry basis is calculated by dividing the wet cake nickel content by the solids fraction.

Three conclusions can be made from the data in Table A-3; (1) the three estimates of insoluble nickel content in the filter cake in each of the six washing tests are substantially the same (although differing from test to test), providing proof that there truly is insoluble nickel in the filter cake; (2) the filter cakes from bench state rejuvenation tests had substantially less insoluble nickel (300 to 600 ppm, dry basis) in the cake from the PU2 fullscale prototype unit (2000 to 3000 ppm of Ni, dry basis); (3) three stage washing is enough to remove substantially all the soluble nickel from filter cake as can be seen by the low values of nickel in the third wash filtrates.

TABLE A-3. INSOLUBLE NICKEL CONTENT OF FILTER CAKES.

Sample I.D.	Cake Solids (percent)	Ni in Wet Cake (ppm)	Ni in Filtrate (ppm)	Insoluble Ni in Cake Wet Basis (ppm)	Insoluble Ni in Cake Dry Basis (ppm)
B1W1C	35.4	800	868	239	676
B1W2C	27.9	300	194	160	574
B1W3C	35.2	300	103	233	662
B2W1C	42.8	746	839	266	622
B2W2C	38.8	326	192	208	538
B2W3C	43.0	229	135	152	354
B3W1C	42.0	770	1040	167	397
B3W2C	33.3	328	274	145	436
B3W3C	43.9	214	81	169	384
BST1W1C	60.2	1296	218	1209	2008
BST1W2C	58.5	1236	80	1203	2057
BST1W3C	57.6	1134	11	1129	1962
BST2W1C	52.3	1773	279	1640	3135
BST2W2C	54.2	1581	94	1538	2838
BST2W3C	62.7	1494	22.5	1486	2369
BST3W1C	55.0	1300	252	1187	2157
BST3W2C	49.9	1157	58	1128	2259
BST3W3C	52.5	1044	29	1030	1962

One of the reasons for filter cakes from the fullscale prototype unit having higher insoluble nickel content was probably that the benchscale tests are conducted in a fresh EN bath and the fullscale tests were conducted with EN solution from a 1-year old bath with an age equivalent of 40 metal turnovers. Hence, the aged bath may be deficient in complexors to hold the nickel in solution. More importantly, filtration times on the fullscale unit were inordinately long (70 minutes versus 15 minutes in benchscale tests) due to an unoptimized filter membrane selection. During filtration, the treated bath solution is at a high pH (8.5 or higher) which promotes nickel deposition on the cake during filtration. Since the filter membrane on the fullscale unit has been changed to complete filtration 15 minutes this problem will not be an issue during demonstration and furthermore demonstration will have a fresh EN bath.

In addition to estimating insoluble nickel content in the cake, the TCLP test results have been correlated with cake nickel content to estimate the extent of nickel "pickup" by the TCLP extractant. During TCLP extraction an aqueous solution equal to twenty times the volume of the cake is used to extract nickel. The extractant "picksup" all the soluble nickel (from the liquid adhering to the cake) and dissolves some of the insoluble nickel. The extent of insoluble nickel "pickup" can be estimated from the nickel content of the filtrate and the cake, cake solids fraction and TCLP test result.

The estimates of the percent "pickup" of the insoluble nickel from the cake are given in Table A-4. As shown, the TCLP extractant picks-up 15 to 30 percent of the insoluble nickel from the cake.

TABLE A-4. ESTIMATE OF PERCENT PICKUP OF NICKEL IN TCLP

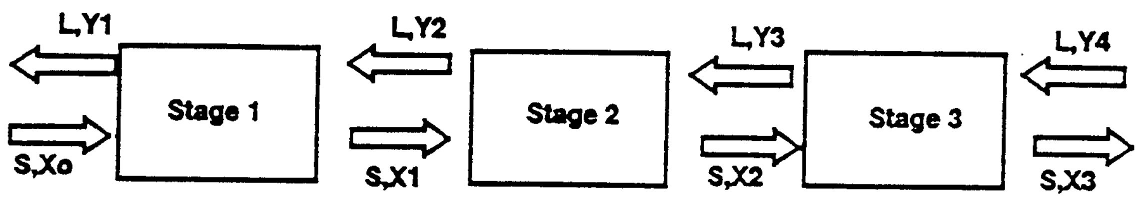
Sample I.D.	Cake Solids (percent)	Ni in Filtrate (ppm)	Ni in Cake*, Dry Basis (ppm)	TCLP Test Value of Ni (ppm)	% Pick-up by TCLP Extractant
B1W3C	35.2	103	610	6.32	30
B2W3C	43.0	135	505	6.66	23
B3W3C	43.9	81	405	4.19	28
BST1W3C	57.6	11	2009	9.56	16
BST2W3C	62.7	22.5	2780	17.10	19
BST3W3C	52.5	29	2126	13.2	23

* Nickel in the cake (dry basis) is the average of the three values of insoluble nickel (dry basis) from the three water washes for each test.

Based on the above analysis of data on insoluble nickel in the cake, percent pickup by the TCLP extractant, a three-stage countercurrent washing system was modelled to remove the nickel. The countercurrent washing system simulation is described below.

C. THREE STAGE COUNTER CURRENT WASHING SYSTEM

The three-stage counter current washing system assumes that the wash water removes only the soluble nickel (in the liquid adhering to the cake) and the insoluble nickel is left untouched. A percent of the insoluble nickel is dissolved by the TCLP extractant and all the soluble nickel in the cake (after the three stage washing) is also picked-up by the TCLP extractant. A model of the washing system is shown below. S and L represent the wet cake and wash liquid flowrates respectively.



X and Y represent the concentration (ppm) of soluble nickel in L and S. If the liquid to solid ratio (L/S) is denoted by A, then the mass balances are:

$$Xo + AY_2 = X_1 + AY_1$$
$$X_1 + AY_3 = X_2 + AY_2$$
$$X_2 + AY_4 = X_3 + AY_3$$

Assumptions are:

(1) Flow rates of L and S are constant from stage to stage

(2) Entering fresh wash water has zero nickel content ($Y_4 = o$)

(3) Liquid adhering to the solids leaving a stage has the same nickel content as the wash liquid leaving that stage. This implies that $X_i = WY_i$ where W is the moisture content of the solids phase.

The above equations can be solved to yield

$$Y_1 = Xo\ [(A+W)^2\text{-}AW]/[(A+W)(A^2+W^2)]$$
$$X_3 = WY_3 = (W^3\ Xo)/(A+W)(A^2+W^2)]$$

Using the above results, simulation of a three-stage countercurrent washing system was performed for various values of nickel in the cake after the rejuvenation filtration (Xo). The expected values of soluble nickel in the cake after three stage washing (X_3) are given in Table A-5. Since the initial cake had nickel content varying from 2000 ppm to 4000 ppm (total nickel, wet basis, see Table A-1), that range was chosen for Xo. In addition, the liquid-to-solid mass ratio was fixed at 3 and the solid fraction of the cake was varied from 0.4 to 0.6 based on test results (see Table A-1). The insoluble nickel content was fixed at 500 ppm, resulting in a soluble nickel content range of 1500 ppm to 3500 ppm. In addition, the expected TCLP test values for nickel for the cakes after three stage washing is estimated. Based on our test results, 25 percent of insoluble nickel is assumed to be picked up by the TCLP extractant (see Table A-4) and 20 to 1 dilution in nickel concentration is assumed for TCLP tests because the extractant weight is twenty times the filter cake weight as per TCLP test protocols.

As shown in Table A-5, a three stage countercurrent washing system will remove more than 99 percent of the soluble nickel (X_3/Xo) and the soluble nickel contribution to TCLP test value is small. As long as the insoluble nickel content of the cake is 500 ppm or less, a three stage countercurrent washing system will render the cake nonhazardous as per the TCLP limit of 5 ppm. If the cake has insoluble nickel content much higher than 500 ppm, then acid washing is the alternative method to render the cake nonhazardous (see Table A-2, Tests 7 and 8).

D. CONCLUSIONS ON CAKE-WASHING

Based on the filter cake-washing tests and subsequent analysis of the test data, the following conclusions can be made.

(1) The cake has soluble nickel and insoluble nickel. The insoluble nickel content increases with filtration times.

TABLE A-5. SIMULATION OF THREE-STAGE WASHING AND EXPECTED TCLP RESULTS.

Liquid to Solid Ratio, W	Liquid Fraction, A	Insoluble Nickel (ppm)	Soluble Nickel			Soluble Ni Contribution to TCLP (ppm)	Insoluble Ni* Contribution to TCLP (ppm)	Total Ni Expected in TCLP (ppm)
			Xo (ppm)	X_3 (ppm)	Y_i (ppm)			
3	0.60	500	1500	9.6	497	0.5	2.5	3.0
3	0.50	500	1500	5.8	498	0.3	3.1	3.4
3	0.40	500	1500	3.1	499	0.2	3.7	3.9
3	0.60	500	2500	16.0	828	0.8	2.5	3.3
3.	0.50	500	2500	9.7	830	0.5	3.1	3.6
3	0.40	500	2500	5.1	832	0.3	3.7	4.0
3	0.60	500	3500	22.4	1159	1.1	2.5	3.6
3	0.50	500	3500	13.5	1162	0.7	3.1	3.8
3	0.40	500	3500	7.2	1164	0.4	3.7	4.1

* Based on dissolution of 25 percent of insoluble nickel in the TCLP extractant.

(2) The soluble nickel part can be washed and removed to achieve a TCLP value of 5 ppm by a three stage countercurrent washing system and the soluble nickel contribution to TCLP extraction is minimal ($<$ 1 ppm) after the three stage washing.

(3) As long as the insoluble nickel content is less than 500 ppm (wet basis) in the cake, the cake can be rendered nonhazardous by three-stage washing, assuming a TCLP value of 5 ppm will be required in the future when EPA establish guidelines for nickel.

(4) If the insoluble nickel content is substantially higher than 500 ppm (wet basis), acid washing will remove the nickel and the cake can then be classified as nonhazardous.